CHEMICAL LABORATORY EXPERIMENTS FOR
(EP 103 / EG 102)

LABORATORY MANUAL

Lecturer/Instructor:

Name: ______________________________

Student ID: ______________________________

Course: ______________________________

FACULTY OF ENGINEERING, ARCHITECTURE & BUILT ENVIRONMENT

UCSI UNIVERSITY 2010

TABLE OF CONTENT

General Instructions

Experimental Illustrating Technique

Experiment 1 Thin Layer Chromatography: Separation Of Organic and Inorganic Dyes

Experiment 2 Separation Of Selected Flammable Compounds Using Gas Chromatography

Experiment 3 Determining The Enthalpy Of Vaporization

Experiment 4 Determining Rate of Reaction by the Effect of Concentration and Catalyst

Experiment 5 Electrochemical: Faraday's Law of Electrolysis

Experiment 6 Thermocouple Calibration

Experiment 7 Investigation of Oxygen Solubility in Water

Experiment 8 Synthesis and Identification of Aspirin

Experiment 9 Synthetic Organic Chemistry

Experiment 10 Determination of Concentration of Potassium Dichromate solution by UV-VIS Absorption Spectroscopy

Experiment 11 Infrared Spectra of Organic Compounds

APPENDIX

A Example of Lab Report Cover Page

B Reference Citing Method

C Lab Promissory Note

GENERAL INSTRUCTIONS

Laboratory experiments are designed with the intention of being completed during the allotted classroom time. In order to expedite things it is mandatory that you be prepared prior coming to the lab. This includes reading the lab procedure handout thoroughly and understanding the experiments illustrations and techniques. In additional, any applicable calculations (such as solution preparations) should be made in advance so that work may commence upon arriving in the lab. A flowchart of the experiment methodology is helpful in determining what steps should be taken and the order in which things must be done. In addition, it is important that you familiarize yourself with lab techniques learned in your chemistry classes.

Some experiments may take longer that the others, but the biggest determinant will be your preparation.

ATTENDANCE IS MANDATORY!

(A) Laboratory logbook

You are required to have a logbook. This must be a bound logbook. It is very important to get into a habit of writing a logbook. You are required to translate the procedure of each lab in the lab manual into graphical illustration or flowchart in your logbook before you begin your experiment.

Recording in the logbook should be written as the experiment is carried out and should be as brief, but concise, as possible. Record in your logbook everything you do and observe as you carry out the experiment.

Each experiment in the logbook should contain the following information:

(i) Title of the experiment
(ii) Date of the experiment carried out
(iii) Objective of the experiment
(iv) Hypothesis of the experiment
(v) Procedure outline or brief description of the method
(vi) Observation (should be recorded throughout the experiment)

A logbook should be neat. Clarification of procedure and results are important. Always use a pen to write your logbook. For any mistake or changing of the data written on the logbook, the usage of liquid paper or eraser is not allowed. Always use a pen to cross out if any errors.

The lab instructor/lecturer will inspect and sign your logbook during the lab session and possibly grade them at the end of the semester.

(B) Laboratory group

You will be assigned to a lab group (please refer to the student notice board or outside the lab after the first session of the laboratory meet up). Students are not allowed to change their team member for lab. You are required to work with you group members as much as possible in terms of understanding the lab work and sharing information on the different sections that were performed by different members. At the end of each of the session, each of the group members will have to evaluate their peers, which will be encounter during the report marking by lab instructor/lecturer. Peer's evaluation is private and confidential. The purpose of peer's evaluation is to ensure all the group members contribute throughout the lab sessions, to avoid any horse riding.

(C) Laboratory reports

i) Report submission

Report must be submitted one week after the scheduled experiment and within the 1st 10 minutes of the due lab period. Marks would be deducted accordingly for late reports or may not be accepted.

ii) Report style/format

Lab report must be in hand written or typed. Each of the group members must contribute in the report. Report should contain the following:

Front page (refer the at Appendices A)
Table of content
Objective

Introduction/background of the experiment
Material and Methodology
Results and discussion
Conclusion
Limitation of the Experiment
Reference
Appendix (if any)

Lab reports are to be assembled in a logical and orderly way. Tedious calculations must be placed in sequential order in the report. The report shall be:

(i) Written in passive tense.
Example: "Five milligram of solid was weighted."

(ii) Graph must be with title, axis labels with units and legend when necessary. The scale of the graph should be adjusted to fit the graph data.

(iii) Plagiarism will not be allowed and tolerated.

(iv) Use suitable analysis method during explanations for the graph such as R-value, slope of the line, line equations, etc in explaining the experiment.

(v) Only chemical compound structures can be drawn using handwriting.

Report outlines and marks distributions:

Introduction/ Background	3%	Contain a concise summary of the experiment that is carried out. Includes the statement of purpose, an introduction into technique used and a brief overview of the instrumentation.
Material & Methodology	3%	Contains all relevant experimental procedures, materials and instrument parameters used in the analysis or during the experiment.
Results & Discussion	5%	-Show all the results from the experiment by using graph, diagram, table, etc. All the data must be at appropriate units. The calculation should shows in systematic basis. Show only one example calculation for repeat data trials. Include statistical analysis if any. All results must be expressed to correct significant figures and in correct units. -Explanation of the figures or graph or data should followed by supporting reference or relevant/logical analysis.
Conclusion	2%	Conclusion should reflect an understanding of the chemical theory involved and achievement of the experiment objective.
Limitation of the experiment	2%	It may include the limitation of the technique or methodology of the experiment based on literature study. Includes also any error that encounter during the experiment is carried out. Suggest for improvement to avoid error.
Reference	3%	A minimum of 3 references should be cited in the experiment report.
Format	2%	Front page, table of content, reference writing style, graph, figure, table, etc.

(D) Laboratory Assessment

At the end of the experiment, there are 5 experiments need to be carried out by each of the students. Below is the assessment of the laboratory.

Experiments (20 each)	100	8%
Peer's rating		2%
Total		10% of the foundation of chemistry mark

(E) Laboratory Rules

All the laboratory rules already learned will be applicable all the time and the students are required to comply with all the rules when there are in the lab.

1. Wear closed shoes, lab coat and eye protection (goggles) at all the time while in the laboratory.
2. DO NOT wear bulky clothes or loose sleeves and long pants. Wear suitable attire for lab work. Those with long hair do tie it before entering the lab. *(lecturer/instructor preserve the right to not allow student who not follow this rule from enter the lab)*
3. Switch off your hand phones.
4. Always be punctual. Students who come later than 15 minutes will not allowed to perform the experiment.
5. Eating and drinking in the lab are strictly prohibited in the lab.
6. Students must have a towel (for cleaning purposes), a logbook and their lab manual during the lab session.
7. Clean your bench and return all the apparatus to their respective places before you leave the lab.
8. Read the lab manual and understand the lab before you going to the lab.
9. Use only the amount of the reagents required. Avoid wastage.
10. Dispose of excess reagents as instructed, never return excess reagents into the bottle.
11. Make sure you know where the following safety devices are: eyewash station, emergency shower, fire extinguisher, fire alarm station.
12. Clean up the broken glass and report to the lab instructor or lecturer immediately.
13. Always follow the instruction of the lab instructor or lecturer before start the lab.

14. Do not casually dispose chemicals into the sink. Some chemicals may washed down the sink, while others may require a different method of disposal. If a chemical can go in the sink, be sure to wash it away rather than risk an unexpected reaction between chemical 'leftovers' later.

EXPERIMENT 1: THIN LAYER CHROMATOGRAPHY: SEPARATION OF ORGANIC AND INORGANIC DYES

OBJECTIVES

The students will observe paper and thin layer chromatography and identify the principles of both.

INTRODUCTION

Chromatography is a technique that can be used to separate and identify a wide range of organic compounds. The separation of compounds in a mixture is governed by the compound tendency to associate with the mobile (usually hydrophobic) liquid phase or to adsorb onto the solid (usually hydrophilic) surface. Three types of chromatography are routinely used:

- Column Chromatography
- Thin Layer Chromatography (TLC)
- Gas Chromatography (GC)

In TLC, the mixture to be separated is first applied as a spot or a line to the solid phase, and then the mobile solvent is allowed to pass through the applied compounds along the stationary phase. The compounds will dissolve in and move with the solvent; the movement of the compounds with the solvent along the stationary phase is called the 'development' of the chromatography.

The separate components adsorbed onto the stationary phase appear as a series of spots at different locations along the plate as shown in Figure 1. The spots can be visualized directly, observed under UV light or sprayed with a reagent to made them visible. The distance traveled by the particular compound is characteristic for a specific set of conditions, such as solvent polarity or pH; and may be used to identify compound. The ratio of the distance traveled by a compound to that of the solvent front is known as Rf value; unknown compound may be identified by comparing their Rf to the Rf of the unknown standards,

$$R_f = \frac{Distance\ \ traveled\ \ by\ \ each\ \ compound}{Distance\ \ traveled\ \ by\ \ mobile\ \ phase} = \frac{dx}{ds}$$

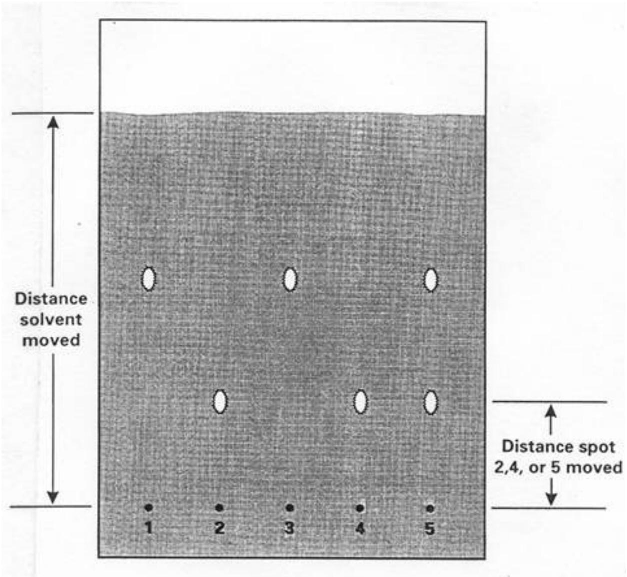


Figure 1: Sample chromatogram

For example, if a compound travels 2.1 cm and the solvent front travels 2.8 cm, the Rf is 0.75:

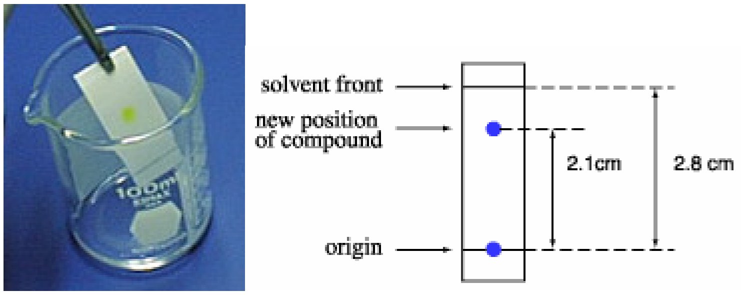


Figure 2 (a) & (b): Illustration of chromatogram development

The Rf for a compound is a constant from one experiment to the next only if the chromatography conditions below are also constant:

- solvent system
- adsorbent
- thickness of the adsorbent
- amount of material spotted
- temperature

A desirable Rf value lies between 0.3 and 0.7, since it is likely that other compounds present in the mixture will be visible on the TLC plate when the Rf is in this range.

MATERIAL AND APPARATUS

Filter paper, beakers, glass watch, capillary tubes, Three TLC plates, TLC developing chamber, n-hexane, ethyl alcohol, toluene, organic dye, inorganic dye

PROCEDURE:

Part I: Procedure for pigments in green plants.

1. Cut away the stems and midribs from fresh spinach and chop into small pieces.
2. Place 2 to 3 grams of the spinach into a small beaker and add 10 ml of acetone.
3. Stir the mixture with a glass rod and crush against the side of the beaker.
4. Pour off the acetone into a clean beaker and then add 10 ml of ethyl alcohol to the spinach.
5. Repeat step four with the alcohol.
6. Pour off the alcohol into the beaker with the acetone. This solution will be your sample for the chromatography of green plant pigments.

Part II: Developing Chromatogram

1. First, using a pencil and ruler, gently draw a line across the bottom edge of each plate, 1.0cm up from the bottom.(only touch the TLC plate at the edges)
2. Make two dots of equal distance on the line. Dip a capillary tube into the organic dye and spot the 1st dot on each of the three plates. In generally, you should keep the spots as small as possible. Let the spot dry before reapplying more dye. Do not mix up the capillary tube between different dyes. Repeat spotting for inorganic dye on the 2nd dot. Do this for all three plates.
3. Place the TLC plate gently into the developing chamber, which fill with different solvent individually. Be careful not to let the solvent level get above the line at the bottom of the plate. Place the cover on top of the developing chamber.
4. Wait and watch the solvent front travel up the plate. When the solvent front has gotten 0.5-1.0cm from the top of the plate, pull it out and immediately mark the solvent front with a pencil line.

5. Observe the separated component spots under UV light and outline them using a pencil. Measure dx and determine the Rf value for the standards. Bsed on the standard Rf values and separation profile, evaluate and comment on the solvent mixture in terms of their suitability for separating the standards.
6. Report stationary phase, moving phase, and Rf for each dye.

RESULTS AND DISCUSSION

In your results and discussion, you should include the below:

1. Why must you use lead pencil, instead of a pen, to mark your chromatography paper?
2. The R_f value for the different solvent mixture on the chromatogram.
3. If two different substances have the same or nearly the same R_f values, it is difficult to separate (resolve) these materials in a mixture. How could this experiment be changed to separate such a mixture?
4. Give two or three practical examples where chromatography would be valuable analytical tool.
5. determine the factors that affect the rate of elution of organic compounds.
6. In order to identify a compound using TLC, a sample of the pure compound must be spotted on the same plate as the unknown. Why is this necessary?

CONCLUSION

State you conclusion of this experiment.

REFERENCE

EXPERIMENT 2: SEPARATION OF SELECTED FLAMMABLE COMPOUNDS USING GAS CHROMATOGRAPHY

OBJECTIVES

- To analyze volatile organic liquids and determine purity of samples
- To identify organic substance qualitatively and determine composition of known compound in an unknown mixtures

INTRODUCTION

Gas chromatography (GC) is an analytical separation technique in which a mixture is vaporized and injected onto a chromatographic column. The inert carrier gas, often helium or hydrogen, is used to transport the components of the mixture through the length of the column. The component of higher boiling point has the longest retention time. Gas chromatography is generally used for the separation of gases and volatile compounds such as organic compounds, essential oil, and petroleum products.

The distribution of analytes between phases can often be described quite simply. An analyte is in equilibrium between the two phases;

$$A_{mobile} \rightleftharpoons A_{stationary}$$

The equilibrium constant, K, is termed the *partition coefficient*; defined as the molar concentration of analyte in the stationary phase divided by the molar concentration of the analyte in the mobile phase.

The time between sample injection and an analyte peak reaching a detector at the end of the column is termed the *retention time* (t_R). Each analyte in a sample will have a different retention time. The time taken for the mobile phase to pass through the column is called t_M.

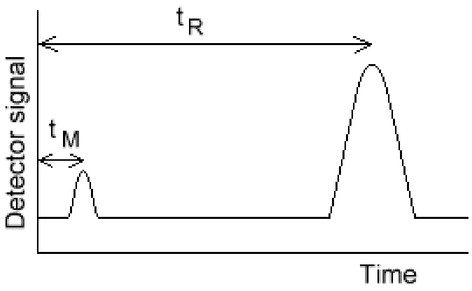


Figure 3: Retention time at the output of the GC

A term called the *retention factor, k'*, is often used to describe the migration rate of an analyte on a column. You may also find it called the *capacity factor*. The retention factor for analyte *A* is defined as;

$$k'_A = t_R - t_M / t_M$$

t_R and t_M are easily obtained from a chromatogram. When an analytes retention factor is less than one, elution is so fast that accurate determination of the retention time is very difficult. High retention factors (greater than 20) mean that elution takes a very long time. Ideally, the retention factor for an analyte is between one and five.

Isothermal gas chromatography involved chromatographic analysis at a constant temperature, is used to separate of simple mixture. Isothermal gas chromatographic analysis was used in the initial study of the identification of petroleum components.

MATERIAL AND APPARATUS

Gas chromatography, capillary column, syringe, solvent, sample, unknown mixture

PROCEDURE

1. Gas chromatography setting (parameters):

    Flow rate of carrier gas (H_2) : mL/minute
    Injection temperature :
    Detect temperature :
    Oven temperature :

2. Determination of t_R

    i. Inject 1.0 μL of the sample mixture. Identify each peak and comment on the shape of the peaks.

    ii. Plot log t_R versus the sample components. Comment on the graph.

3. Analysis of the unknown samples

    i. Inject 1.0 μL of an unknown sample. Comment on the chromatogram. Identify peaks that coincide with t_R of standard peak.

    ii. To confirm the peak, add a drop of particular standard into the unknown and re-inject the mixture. The particular peak should be increase in its area. From the chromatogram, identify as many components as you can with reference to their retention time.

RESULTS AND DISCUSSION

In your results and discussion, you should include the below:

1. Determination of t_R
2. Analysis of the unknown samples based on the output result
3. Answer the self-assessment questions as shown on the poster.

CONCLUSION

State your conclusion of this experiment.

REFERENCE

EXPERIMENT 3: DETERMINING THE ENTHALPY OF VAPORIZATION

OBJECTIVE

Student will experimentally measure the vapor pressure of water as a function of temperature change, prepare a graph of ln(P) as a function of $\frac{1}{T(K)}$ and calculate the enthalpy of Vaporization (ΔH vap) of water.

INTRODUCTION

When vapor is considered as an ideal gas, the change in its saturated vapor pressure, P, against temperature is given bu the *Clausius-Clapeyron* equation:

$$\frac{d \ln P}{dT} = \frac{\Delta H_v}{RT^2}$$

ΔH_v : molar enthalpy of vaporization.

If P is measured in atmosphere, and assuming that ΔH_v do not depend on temperature, the equation can be integrated giving:

$$\ln P = \frac{\Delta H_v}{R}\left(\frac{1}{T_b} - \frac{1}{T}\right)$$

T_b: normal boiling poit at pressure P=1 atm.

MATERIAL AND APPARATUS

Beaker, graduated cylinder, thermometer, ruler, ice, Bunsen burner (or electrical heater)

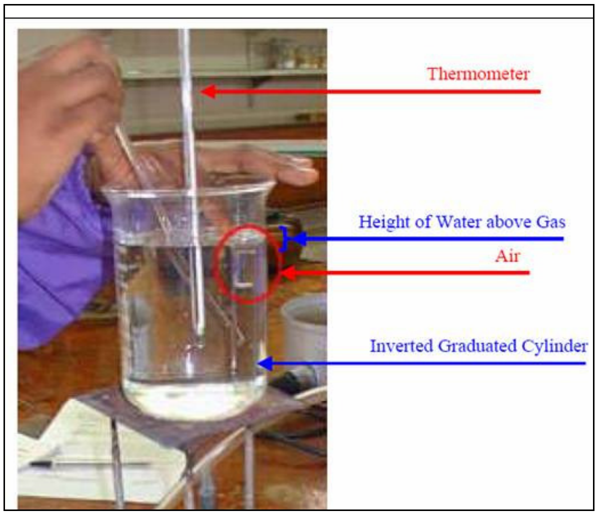


Figure 4: Illustrating of equipment set up

PROCEDURE

1. Obtain a 10 mL graduated cylinder and fill it with approximately 9 mL of water.
2. Fill a 1000 mL beaker approximately three-fourths full with water.
3. Cover the top of the graduated cylinder with your finger and invert it into 1000 mL beaker. Do not release your finger until the mouth of the graduated cylinder is under the surface of the water in the beaker.
4. If the graduated cylinder is not yet covered with water, add water to 1000 mL beaker to cover it.
5. Use a ruler and measure the different between the height of the water in graduated cylinder and the height of the water in the beaker. This will provide a slight adjustment for the pressure that the water exert on the air in the graduated cylinder.
6. Record the barometric pressure in mmHg.
7. Heat the 1000 mL beaker on Bunsen burner (or electrical heater) until the water is about 80°C. If the air in the cylinder expands past the scale on the graduated cylinder, the experiment will have to be restarted using a smaller amount of air.
8. Record the temperature and the volume.
9. Cool the beaker until the temperature reaches 50°C, recording the temperature and volume every 5°C.

10. Once the beaker reach 50°C, cool the beaker rapidly to about 0°C by adding ice. Record the gas volume and the temperature at this low temperature. At this temperature, if the vapor pressure is so low that it can be assumed that all of the gas in the graduated cylinder is air.

RESULTS AND DISCUSSION

In your results and discussion, you should include the below:

1. Plot of the experimental data in a form ln (P) vs. $1/T$, evaluate the slope and calculate ΔH_{vap} for the substances studied; you can collect them in one graph. Identify sources of error and estimate uncertainties.

2. Compare ΔH_{vap} with literature values. Comment on deviations of your experimental value from the literature value.

CALCULATIONS

The 10 mL cylinder is graduated to give correct volume readings only when right side up. This is due to the shape of the water meniscus. Subtract 0.2 mL from each of your readings to correct for this effect.

2. Calculate the total pressure of the gas in the cylinder from the atmospheric pressure and the difference in water levels between the top of the water in the beaker and the top of the water inside the cylinder, which is denoted as h.

$$P_T = P_{atm} + h \text{ mmH}_2\text{O} \left(\frac{1.00 mmHg}{13.6 mmH_2O} \right)$$

3. Calculate the moles of trapped air by using the volume of air present at the lowest temperature. At this low temperature, the vapor pressure of water is negligible.
   $P_TV=n_{air}RT$
4. For each temperature above the lowest one, calculate the partial pressure of air in the cylinder, using the constant moles of gas calculated in # 3. $P_{air}V=n_{air}RT$
5. Calculate the vapor pressure of water at each temperature. $P_{water}= P_T - P_{air}$
6. Plot ln P_{water} on the vertical axis versus 1/T on the horizontal axis.

7. Calculate the value of ΔH_{vap}.
8. Calculate the percent error.

EXAMPLE OF RESULT DATA RECORDS

Atmospheric Pressure ___________

Difference in height, *h*___________

Temperature (°C)	Volume (mL)

Total pressure in cylinder, P_T	
Moles of trapped air, n	

Corrected Volume (mL)	Temp (K)	P_{air} (atm)	P_{water} (atm)	1/T (1/K)	ln P_{water}

Regression Eqn	
ΔH_{vap}	
Percent error	

CONCLUSION

State the conclusion of the experiment

REFERENCE

EXPERIMENT 4: DETERMINING RATE OF REACTION BY THE EFFECT OF CONCENTRATION AND CATALYST

INTRODUCTION

In order to be in a position to manipulate any operation, one has to know all the fine details of the system and where and how one might apply subtle changes to affect the outcome. When looking at chemical reactions, it is from the study of its kinetics that one gleams the insight necessary to be able to do this. The initial focus of chemical kinetics is to measure the speed of a chemical reaction. In measuring the speed of a reaction however, one gains insight as to what part of the reaction imparts this speed (or lack thereof) and thus allows one to propose a specific mechanism that details the critical pathway for the reaction. There are many factors that affect the speed of a chemical reaction, some of the more common ones being:

- Concentration Effects
- Temperature Effects
- Surface Area
- Catalysts

Of interest here is the reaction of potassium permanganate with oxalic acid. This is quite a complex oxidation reduction reaction that you have already encountered when you analyzed $K_3[Fe(C_2O_4)_3].3H_2O$ in experiment two.. The nice thing about this reaction is that potassium permanganate is a deep purple color but when it has been consumed, it turns a light brown and thus this reaction can be monitored visually. If, as we propose, the speed (rate) of this reaction is dependent on the concentration of the reactants then we could write:

Rate is proportional to $[KMnO_4]^x[H_2C_2O_4]^y$

or

Rate = $k[KMnO_4]^x[H_2C_2O_4]^y$

This is called the rate equation, with the sum of the exponents x and y referred to as the order of the reaction. At this introductory level to kinetics, the order of the reactions chosen is usually 0, 1 or 2. However, reactions are not constrained to this and the order can be negative and need not be an integer.

The Rate Method of Initial Rates

In order to determine the speed of the reaction we are going to focus here on the permanganate and simply equate the speed with the time it takes to use up the permanganate.

[KMnO□Rate = -$_4$t = -{[KMnO□]/ $_4$]$_{final}$ - [KMnO$_4$]$_{init.}$]/(t$_{final}$ - t$_{initial}$)

This simplifies quite remarkably as [KMnO$_4$]$_{final}$ = 0, and by setting t$_{initial}$ =0.
Rate = -(-[KMnO$_4$]$_{init}$/t$_{final}$) = [KMnO$_4$]/t

We can then use the method of initial rates to find the exponents x & y. This method involves measuring and comparing the initial rates of a reaction when different initial concentrations are used. The initial rate for each being the potassium permanganate concentration divided by time. There is an inherent assumption made here that is not always valid: we will be measuring t$_{final}$ to determine each rate. If you look at the volume of each reagent added for each of the three experiments, you will notice that experiment 2 and 3 are related to 1 in that one of the concentrations is identical while the other is halved. This helps dramatically with the mathematics involved because if we take the ratio of the rates of reaction 1 with reaction 2 we get
$Rate_1 = k[KMnO_4]_1^x[H_2C_2O_4]_1^y$ $Rate_1 = [KMnO_4]_1/t_1$
$Rate_2 = k[KMnO_4]_2^x[H_2C_2O_4]_2^y$ $Rate_2 = [KMnO_4]_2/t_2$

Now take the ratio:
$Rate_1/Rate_2 = ([KMnO_4]_1/[KMnO_4]_2)^x$
(remember in these two reactions $[H_2C_2O_4]_1 = [H_2C_2O_4]_2$, and thus can be canceled out)

If you do the experiment carefully, it should be apparent what the value of x should be. It should turn out to be a whole number. If you want to find its exact value and thus get a feel for the accuracy of the experiment, then the following general log relationship will be of use:
$\log_{10}[a^x] = x\log_{10}[a]$

Thus we can determine x. If we now do the same thing for experiments 1 and 3 we can get y. Once we know what x and y are, then by taking any one of the three experiment we can solve for k, the rate constant. The above may sound somewhat cumbersome and difficult, but as you will see when you collect the data it is not difficult at all.

MATERIAL AND APPARATUS

$KMnO_4$ (0.02M), $H_2C_2O_4$(0.05M)
Erlenmeyer flask, burette

PROCEDURE

Part I: Determining the rate of a chemical reaction Effect of concentration

1. Set up three burets, one containing $KMnO_4$ (0.02M), one with $H_2C_2O_4$(0.05M) and one with distilled water. Place a label on each. It is difficult to visually differentiate between water and oxalic acid! Record the exact molarities of the $KMnO_4$ and $H_2C_2O_4$.
2. Into a thoroughly washed and dried Erlenmeyer flask, place the required amount of $H_2C_2O_4$ and any distilled water. The amounts are dictated by the experiment that you are doing. You have used burette sufficient times by now that you should be able to dispense the required amount. If you overshoot, start again. This lab is very dependent on dispensing the exact quantities.

Table 2: Experiment (Part I) reagents mixture composition

Reagents	Exp #1 (mL)	Exp #2 (mL)	Exp #3 (mL)
$H_2C_2O_4$	20.00	20.00	10.00
$KMnO_4$	10.00	5.00	10.00
H_2O	0.00	5.00	10.00

3. Into a 15cm test tube place the required amount of $KMnO_4$.
4. Add the permanganate to the oxalic acid and commence timing when you have emptied the permanganate tube. Mix thoroughly by swirling the Erlenmeyer flask and continue swirling until the solution turns a light yellow/brown color. Stop timing and record the time it actually took for the reaction to take place.
5. Repeat steps 3 through 5 for experiments 2 and 3.
6. Determine the rate for each of the three experiments. Remember this is just the $[KMnO_4]/t_{average}$
7. Write out the full rate equation for each experiment. Again remember that this is equal to: $Rate_{Exp\#x} = k[KMnO_4]^x[H_2C_2O_4]^y$.

Part II: Determining the rate of a chemical reaction Effect of catalyst

1. Conduct the following dilution.
2. $KMnO_4$ – Dilute 50 drops of $KMnO_4$ (0.02M) with distilled water to a volume of 25 mL.
3. $H_2C_2O_4$ – Dilute 50 drops of $H_2C_2O_4$ (0.05M) with distilled water to a volume of 25 mL.
4. Using the solution for the below experiment.

Table 3: Experiment (Part II) reagents mixture composition

Reagents	Exp #1 (mL)	Exp #2 (mL)
$H_2C_2O_4$	20.00	20.00
$KMnO_4$	10.00	5.00
H_2SO_4	0.00	5.00

5. Into a test tube, add the amount of the $H_2C_2O_4$ and $KMnO_4$ and record the time starting from the addition of the $KMnO_4$ solution to the disappearance of the $KMnO_4$ color.
6. Repeat the Exp #2 with adding H_2SO_4. Record the time starting from the addition of the $KMnO_4$ solution to the disappearance of the $KMnO_4$ color.

RESULTS AND DISCUSSION

In your results and discussion, you should include the below:

Part I

Your 'Data Collection' should be tabulated and included:

- The molarity of $KMnO_4$ and $H_2C_2O_4$
- The volumes used.
- The time it took for each trial for each experiment.

Your 'Calculations' section should also include a table that should record

- The concentration of $KMnO_4$ used in each experiment.
- The concentration of $H_2C_2O_4$ used in each experiment.

- The average time for each experiment,
- The initial rate for each experiment.

  (You should show one sample calculation for each of these.)

In your discussion you should summarize your results addressing:

- What was the overall order of this reaction.
- The rate constant for the reaction.
- The order with respect to $KMnO_4$ and $H_2C_2O_4$.

Part II

1. Why does it take so much longer for the color of the solution in the beaker on the left to change?
2. Write a balanced chemical equation for the reaction of permanganate ion and oxalic acid to give Mn^{2+} and carbon dioxide. The reaction takes place in an acidic solution.
3. The reaction in the beaker on the left eventually proceeded at a reasonably rapid rate. Explain how this observation supports a reaction mechanism involving autocatalysis.

CONCLUSION

State the conclusion of the experiment.

REFERENCE

EXPERIMENT 5: ELECTROCHEMICAL: FARADAY'S LAW OF ELECTROLYSIS

OBJECTIVE

To carry out the **electrolysis** with two metals in copper(II) sulfate solution and to link their findings with the industrial **electrolytic refining of copper.**

INTRODUCTION

In chemistry, you might have learnt that some reactions proceed easily (spontaneously) either naturally or on application of heat. Other reactions can only occur *(need to be forced to proceed)* by supplying energy with an externally applied electric current, for example, a battery. This process is called electrolysis.

Electrolysis is important in that it is used in the extraction of reactive metals, such as potassium, sodium, calcium, magnesium and aluminium; electroplating metals to improve their appearance and reduce corrosion/prevent rusting and in refining metals such as copper. For example, copper is mined in Kilembe and is transported to Jinja for purifying electrolytically. Electroplating can be done at home once you have the necessary substances to use and intend to start a small business, for example, coating iron or aluminium bangles with copper.

MATERIAL AND APPARATUS

DC power, 1L beaker, 2 insulated electrical wires with crocodile clips at each end

Electrodes: Metal strips of Tin, Aluminum, Copper

Electrolyte solution: $CuSO_4$ solution, of any molarity

PROCEDURE

Part A. electrolysis of aqueous copper(II) sulfate

1. Obtain both + and – electrodes. Clean them with emery paper, then rinse with distilled water and dry thoroughly.
2. Weigh and record their masses.
3. Fill electrolyte solution about 2/3 in a 1 L beaker.
4. Immerse the Anode and the Cathode by clamping them to the rim of the beaker using the crocodile clips. Make sure the electrodes do not touch each other.
5. Connect the electrodes to the instrument.

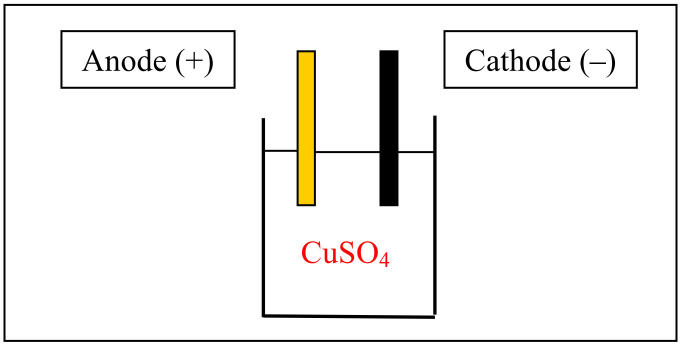


Figure 5: Illustrating of the equipment set up (Exp. 5)

6. Switch on the current, set to allow a current of 0.3 A passes through the solution.
7. After 15 minutes, remove the electrodes from the electrolyte, wash them carefully with distilled water and dry them in oven.
8. Re-weigh the dry electrodes.
9. After about 8 times step 7 being repeated, switch off the current.
10. Calculate the electric charge (coulombs) using Q=It
11. Plot the graph of electric charges in coulombs corresponding to the increase in weight of cathode. From the graph, find the slope of equation.

Part B. Purification of copper electrolytically

1. Thoroughly clean a copper strips using emery paper. Rinse carefully with distilled water.
2. Dry both copper strips and then weighed them.
3. In copper (II) sulphate solution, connect the cleaned copper to negative supply (cathode) and the other strip to positive supply using crocodile clips (**do not immerse clips in the solution at any stage**).

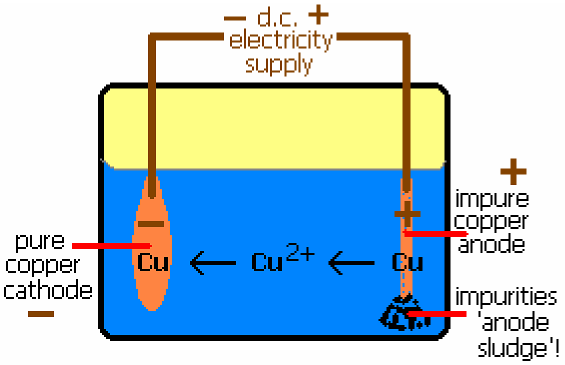


Figure 6: Refining Copper by electrolysis (Exp. 5)

4. Switch on the voltage, run the electrolysis for 15 minutes.
5. Start observing the reaction that is taking place.
6. Carefully remove the electrodes from the solution. Rinse them with distilled water, dry and weigh them again.

RESULTS AND DISCUSSION

EXAMPLE OF TABLE OF DATA

Cathode (-) before electrolysis /g	
Cathode (-) after electrolysis /g	
Change in cathode's mass /g	
Anode (+) before electrolysis /g	
Anode (+) after electrolysis /g	
Change in anode' mass /g	
Current used /A	
Time used /s	

In your results and discussion, you should include the below:

1. Observation of the experiment changes, Reactions at the electrodes in terms of electron transfer. Explain the reactions taking place in view of the ions discharged using dilute sulphuric acid.
2. Voltage and current measurement data during the process.
3. Provide the application of electrolysis separation in industry by example.
4. Assigned Problems:

   In an electrolysis of copper electrode in aqueous copper(II) sulfate solution, 6.35g of copper deposited at the cathode.

   a. Calculate the number of coulombs needed. (Given 1Faraday=96500 coulombs)

   b. Calculate the time that a current of 4 amps must pass to deposit this mass of copper.

CONCLUSION

State the conclusion of the experiment

REFERENCE

EXPERIMENT 6: THERMOCOUPLE CALIBRATION

OBJECTIVE

To calibrate a thermocouple and find the corresponding curve-fit correlation.

THEORY

When two dissimilar metal wire are connected with each other in a loop to form two junctions, maintained at two different temperatures, a voltage potential or electromotive force (E=emf) will be generated and the current will flow through the loop circuit. The current will be proportional to the difference in temperature between the junctions and the metals used. The higher the temperature difference, the higher is the electromotive force (emf) and the current flow in the loop. The magnitude of the emf is in the order of few millivolts. We need a precise and sensitive multimeter (MM) which can read up to microvolts to do this experiment.

MATERIAL AND APPARATUS

1. Thermocouple
2. Beaker
3. Thermometer
4. Multimeter

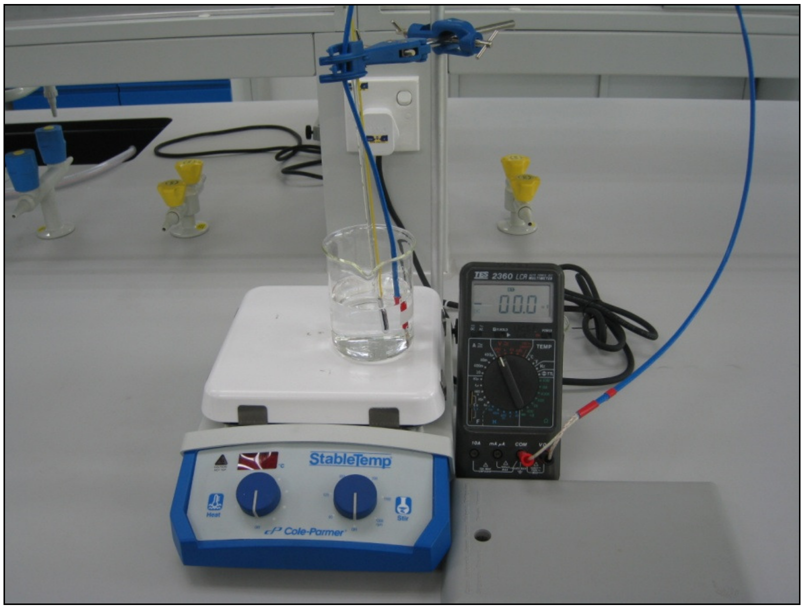


Figure 7: The equipment set up for thermocouple calibration

PROCEDURE

1. Assemble the apparatus as shown in the picture.
2. Turn on the hot plate to increase the temperature of the water in the beaker.
3. Heat the beaker to the desired temperature (30 ° C to start). Switch off the hot plate once the temperature is found.
4. Connect the ends of the thermocouple to the digital multimeter (MM) and set the multimeter to read in millivolts DC.
5. Dip one junction of the thermocouple in the thermo-bath liquid and wait for few minutes for it to reach the steady state (i.e. the reading on the MM steadies down except the last digit). **BE CAREFUL** to hold (tape) the sensor wire away from the circulator's propeller!
6. Note down the digital MM reading in millivolts (E_{MM}) and repeat the steps 3 through 6 in steps of 5 degrees from 30 to 60 degrees Celsius.
7. As mentioned in the theory, the multimeter reading corresponds to the difference in temperature between the surroundings (room) and the bath. To calibrate the thermocouple, we have to take the room temperature into consideration to get the absolute value of temperature measured. Find the equivalent millivolt value for the room temperature from the corresponding Thermocouple table (E_{RM}). Then, add that millivolt value, corresponding to the room temperature, to every multimeter reading (E_{MM}). Tabulate the values.

RESULT AND DISCUSSION

1. Plot the measured bath temperatures values (T_B) on y-axis against the corresponding thermocouple emf (millivolt) values (E) on x-axis.

2. Find the slope, intercept and the correlation coefficient of the curve-fitted line by any method. If the correlation coefficient is not very close to one, curve fit with higher order polynomial. Calculate the 95% uncertainty in temperature measurement of the calibrated thermocouple sensor if the obtained curve-fit correlation is used.

CONCLUSION

State the conclusion of the experiment

REFERENCE

EXPERIMENT 7: INVESTIGATION OF OXYGEN SOLUBILITY IN WATER

INTRODUCTION

This experiment is to illustrate that oxygen dissolves to varying degrees under different conditions.

Have you ever wondered how microorganisms and fish in the water live? Definitely they need to breath, therefore oxygen is essential for their continuous existence.. However, sometimes fishes and other organisms die although still in the water. This is due to the insufficient amount of oxygen in the water. Fish for example, cannot continue living if oxygen concentration drops below 5 ppm.

PROCEDURE

Therefore, today's experiment is about evaluating the solubility of oxygen in 4 different sources of water samples.

1. Tap Water
2. Distilled Water
3. Rain Water
4. Drain Water

Dissolved oxygen (DO) in sample is to be measured at room temperature. Comment on your results.

Take a sample of tap water and measure (DO), then heat the sample about 10 degrees above room temperature and then mix the sample well and measure (DO). Continue heating at intervals of about 10-15 degrees , each time measure (DO). Do not exceed 50 °C.

(DO NOT PUT THE PROBE IN HIGH TEMPERATURE WATER).

Allow the hot water to cool to room temperature with continuous shaking and mixing. Once the temperature reaches about 25 °C, measure (DO).

RESULTS AND DISCUSSION

Plot a graph of dissolved oxygen (mg) against temperature in degrees Celsius.

Comment on the graph. What are the effects of temperature on solubility?
Now blow some air bubble into the water again and take another measurement.
What is the reading now?

Waste water should have the minimum value as (DO) is consumed in oxidation of organic materials present such as oils and fats.

CONCLUSION

State the conclusion of the experiment

REFERENCE

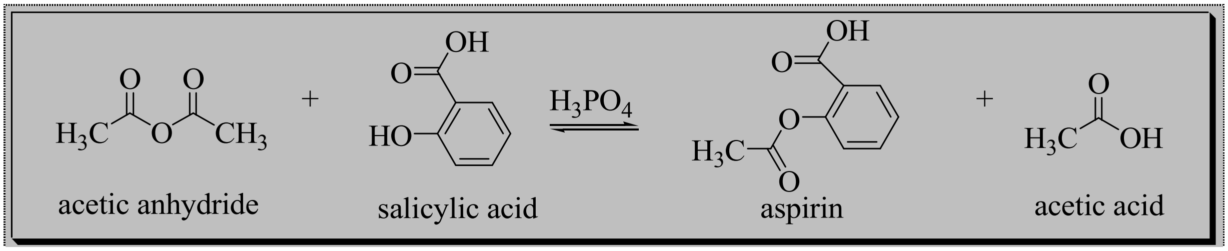
EXPERIMENT 8: SYNTHESIS AND IDENTIFICATION OF ASPIRIN

OBJECTIVE

To explore the chemical process of synthesis by synthesizing aspirin (acetylsalicylic acid)

INTRODUCTION

Aspirin is a relatively simple molecule containing an ethylated phenol group and a carboxylic acid group. In your experiment you will make aspirin from an acid called salicylic acid by esterification with acetic anhydride under acid catalysed conditions. Acetic anhydride is an 'activated' form of ethanoic acid which most of you will have encountered in its dilute form as the vinegar you put on fish and chips. The aspirin will be isolated by filtration and washed several times with ice cold deionized water to remove the water soluble impurities (acetic acid and sulfuric acid). Aspirin is quite insoluble in ice cold water, but is relatively soluble in warm water (1 g dissolves in 100 mL water at 37°C).



During the synthesis steps of the procedure, wear gloves and safety goggles. Both phosphoric acid and acetic anhydride are corrosive and will cause burns if spilled on your skin. Salicylic acid is a skin irritant.

CAUTION!! Concentrated H_2SO_4 is very corrosive! The reaction vessel must be very DRY to ensure a complete reaction.

MATERIAL AND APPARATUS

Balance, buchner funnel, vacuum flask, test tubes, beakers, Elermenyer flask, burner, filter paper, watch glass, acetic anhydride, ice, salicylic acid, concentrated sulfuric acid.

PROCEDURE:

Part A: Synthesis of Aspirin

1. Weigh out approximately 1.0 g of salicylic acid in a 100 mL Elermenyer flask. *Perform the next operation in the fume hood*:
2. Pipet 3.0 mL of acetic anhydride and pour it into the flask in such a way as to wash any crystals of salicylic acid on the walls down to the bottom. Add a few drops of concentrated sulfuric acid to the mixture to serve as a catalyst.
3. Clamp the flask in the water bath, and immerse it in the hot water bath for 5 minutes, stirring the liquid in the flask occasionally with a stirring rod.
4. Once the reaction is complete, remove the flask from the water bath, and CAUTIOUSLY add 10 – 20 drops of water to the mixture to decompose any excess acetic anhydride. There will be some hot acetic acid vapor evolved as a result of the decomposition of any unreacted acetic anhydride.
5. Let the flask cool for a few minutes in air, during which time crystals of aspirin should begin to form.
6. Put the flask in an ice bath to hasten crystallization and increase the yield of product. If crystals are slow to appear, it may be helpful to scratch the inside of the flask with a glass rod.
7. Collect the aspirin by vacuum filtration. Pour distilled water over the crystals; repeat the washing process, and then draw air through the funnel for a few minutes to help dry the crystals. Determine the mass of your impure aspirin.

RESULTS AND DISCUSSION

Molecular Weight of salicylic acid	
No. of moles of salicylic acid	
Molecular Weight of acetic anhydride	
No. of moles of acetic anhydride	
The limiting reagent	
Molecular Weight aspirin	
Theoretical yield	no. of moles of limiting reagent * Molecular weight of the product
	= g
% yield	(actual yield/theory yield) x 100
	= %

Part B: Recrystallization of Crude Aspirin

Recrystallization

Materials that are made for human consumption must be checked thoroughly to ensure that:

- the material is the correct product; and
- it is highly pure.

The aspirin you will prepare in this experiment is somewhat impure. We will attempt to purify the aspirin via recrystallization with ethanol. The purified compound will be characterized by its melting point and by infrared spectroscopy.

CONCLUSION

REFERENCE

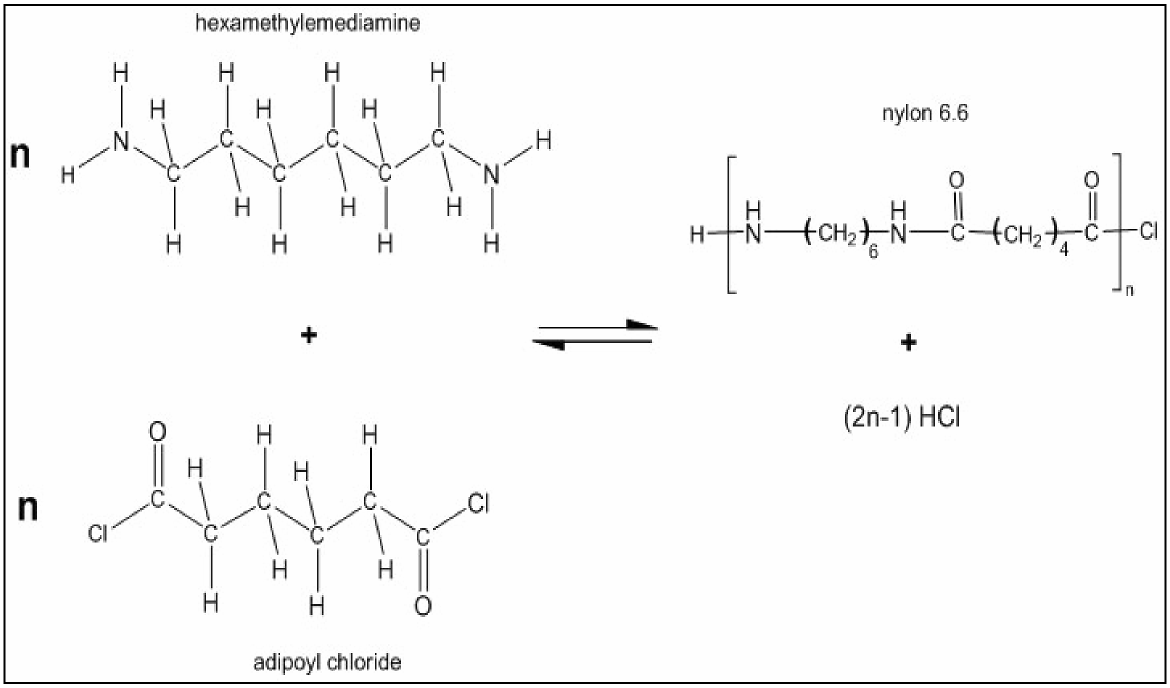
EXPERIMENT 9: SYNTHETIC ORGANIC CHEMISTRY

OBJECTIVE

The objective of this demonstration is to show the formation of a condensation polymer (Nylon)

INTRODUCTION

Nylon is one of many man-made materials called plastics. Chemically, plastics are composed of chain-like molecules of high molecular weight, called polymers, which usually have been built up from simpler chemicals called monomers. A different monomer or combination of monomers is used to manufacture each different type or family of polymers. In this experiment, you will perform the synthesis of nylon. Nylon can be prepared by reacting hexamethylenediamine with adipoyl chloride. For each reaction between the two starting materials, one HCl molecule is split out and an amide bond is formed. Because each of the reactants has two functional groups, one at each end, both molecules react in both directions, and the reaction repeats until a long-chain polyamide results.



MATERIAL AND APPARATUS

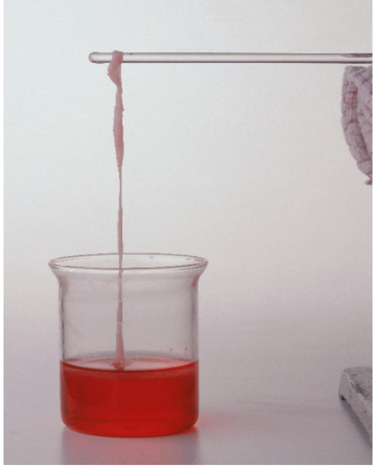
Hexamethylenediamine 70%, sodium hydroxide pellets, adipoyl chloride, cyclohexane, methyl red, distilled water, wooden splint, forceps.

CAUTION: Before starting this experiment make sure that all gas burners have been turned off. Mix all solutions in the open hood. Do not work at your regular bench space until the solutions have been mixed and poured together.

Safety goggles and protective gloves must be worn.

PROCEDURE

1. To one dry 200mL beaker, dissolve 3mL of 70% hexamethylenediamine in 50mL of water, and then add 1.5 g of sodium hydroxide pellets.
2. Stir the mixture until the sodium hydroxide pellets dissolve.
3. Add some methyl red or phenolphthalein to the water solution in the beaker.
4. To another beaker, add 50mL of cyclohexane solution and 2mL of adipoyl chloride, and swirl the flask until the contents are well mixed.
5. By the use of a stirring rod, place the solution containing adipoyl chloride on the top of the solution containing hexamethylenediamine.
6. At the interface between the two solutions you should see nylon begin to form immediately.
7. Carefully carry the beaker from the open hood to your regular working space.
8. Using a pair of forceps, grab the nylon and carefully lift up on the stirring rod. Twisting as you pull, lift a rope of nylon out of the solution.



9. After a little while, the reactants are exhausted and the nylon string getting thinner.
10. Wash the nylon rope with water and then wash it with about 5mL of acetone in a beaker.
11. Weight the final product, examine the rope and record your observations regarding its physical properties on your logbook.

RESULTS AND DISCUSSION

In your results and discussion, you should include the follow:

1. Nylon is classified as a condensation polymer. Describe what is meant by the term condensation polymer and what other product is formed in the reaction producing nylon.
2. Name five common uses for Nylons in general and Nylon 6 in particular.
3. Why are nylon ropes better in many applications than cotton, jute, and metal cables? (A good example is on boats, barges, and ships.)

CONCLUSION

State the conclusion of the experiment

REFERENCE

EXPERIMENT10: DETERMINATION OF CONCENTRATION OF POTASSIUM DICHROMATE SOLUTION BY UV-VIS ABSORPTION SPECTROSCOPY

OBJECTIVES

1. To determine the wavelength (color) of maximum absorbance, λ_{max} for a potassium dichromate solution.
2. To examine the relationship between the absorbance and concentration of a potassium dichromate solution.
3. To prepare and run a standard calibration curve, and determine the concentration of an unknown potassium dichromate solution.

INTRODUCTION

Absorption of light by solution is one of the oldest and still one of the more useful instrumental methods. The wavelength of light that a compound will absorb is characteristic of its chemical structure. Absorption of visible and ultraviolet (UV) radiation is associated with excitation of electrons, in both atoms and molecules, to higher energy states. Because absorption spectra are characteristic of molecular structure, they can be used to qualitatively identify atomic and molecular species. The amount of light, I, transmitted through a solution of an absorbing chemical in a transparent solvent can be related to its concentration by Beers Law:

$$\log_{10}\frac{I_0}{I} = A = \varepsilon\, bc$$

where I_0 is the incident light intensity, A is the absorbance (a defined quantity, also referred to as the optical density, or OD), b is the cell path length in cm, c is the solution concentration in moles/liter, and ε is the molar absorptivity, (also referred to as the molar extinction coefficient) which has units of liter/mole/cm (i.e., A is a dimensionless quantity). Notice that ε is a function of wavelength, and it is the quantity that represents the spectrum of the solution. When its value is stated, it must be stated for a particular wavelength. The only exception to this statement is when its value at the peak of the spectrum is given, in that case, it may be denoted as max. Thus, absorption spectroscopy can be used to quantify the amount of chemical present in an unknown solution.

This experiment demonstrates the principles involved in the spectrophotometric determination of coloured ion, i.e., dichromate ion, $Cr_2O_7^{-2}$, which have an intense orange color. The absorption spectrum of the potassium dichromate is determined within the wavelengths range of 200-600 nm. The quantitative relationship between the amounts of light absorbed by a sample and the concentration of that species will be studied. This relationship is defined by Beer's law, which has been discussed. The absorbances of a series of standard dichromate solutions will be measured at the wavelength found by the absorption spectrum. To evaluate linearity, plot a graph of concentration against absorbance for the maximum wavelengths.

MATERIAL AND APPARATUS

Pipet, pipet pump, 50mL and 100mL volumetric flasks, beakers, analytical balance

Distilled water, potassium dichromate, unknown potassium dichromate solution

PROCEDURE

Preliminary Preparations

Turn the instrument so that the light sources can warm up and stabilize. * (This is done by your laboratory tutor) Next prepare or gather all of the samples that you will need to perform the experiment.

Part A. Stock solution of Chromium (100ppm)

Use 100mL volumetric flask to prepare a Cr stock solution by pipeting 4.9 mL of potassium dichromate (0.02mol/L) and diluted it to the mark with distilled water.

Part B. Preparation of Calibration Solutions of Cr

1. Use 50 ml volumetric flasks to prepare five standard solutions with concentration 2.5ppm, 5ppm, 7.5ppm, 10ppm and 12.5ppm prepared from stock solution which prepared in part I.
2. The mixture is diluted to 100mL with distilled water. Dilution is done by using pipette.

Table 1

Concentration (ppm)	Volume need to be pipette (mL)
2.5	
5.0	
7.5	
10.0	
12.5	

(*Note: If you are unsure of how to read the pipets, please ask. The most common mistake in this lab involves measurements with the pipets. Any time the pipet is used with a new solution, rinse it with that solution before measuring)*

Part C. Measurement

1. The absorbance of the standard solutions is measured by using distilled water as blank. First prepare a *blank* by filling a cuvette 3/4 full with distilled water.
2. Measure the absorbance for each of the solutions in *Table 1*, by placing them into the colorimeter, aligning the reference marks, closing the lid, and determine the wavelength of the maximum absorbance (λ_{max}) as well as their absorbances.
3. Record the results on your spreadsheet. Make sure to include the value of the wavelength at which you are making your measurements.
4. Using *Excel*, prepare a graph that plots absorbance vs. concentration. Obtain the equation of the best-fit line, setting the *y*-intercept to zero. Also, determine the correlation coefficient (r^2) in this linear regression equation.

   (Think: why should the y-intercept be set to zero? What does the slope represent in Beer's Law?)
5. Fill a cuvette about 3/4 full with your unknown sample of potassium dichromate. *Record your unknown number*. Measure the absorbance of the unknown as before. *If the absorbance is greater than any of the measurements used to prepare your trendline, dilute the solution and measure the absorbance again. Take note of the volumes used in your dilution.*
6. Use Beer's Law and the equation of your best-fit line to determine the concentration of the unknown solution. *If you diluted your unknown, calculate the concentration of the undiluted solution and report the result.* Clearly label the unknown number and its concentration.
7. Discard the solutions as directed by you tutor. Clean the cuvettes.

(Handling cuvette: Cuvettes should be wiped clean and dry on the outside before each measurement. Handle the cuvettes near the top of the ribbed sides. Solutions should be free of bubbles.)

RESULTS AND DISCUSSION

Wavelength of maximum absorbance ________________________________

Unknown ________________________________

Dichromate concentration (ppm)	Absorbance, A
2.5	
5.0	
7.5	
10.0	
12.5	
unknown	

Concentration of unknown dichromate solution

- What is the Beer-Lambert law?
- What is a blank? When is it recorded? Why is it recorded?
- What is the typical energy range of light for UV/VIS spectroscopy?

CONCLUSION

REFERENCE

EXPERIMENT11: INFRARED SPECTRA OF ORGANIC COMPOUNDS

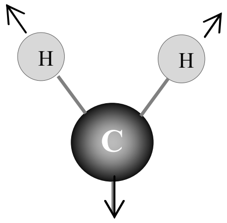
OBJECTIVES

- To learn the basic principles of vibrational spectroscopy
- To determine the relationship between molecular structural features and absorptions in the IR spectrum
- Obtain and interpret IR spectra of organic compounds

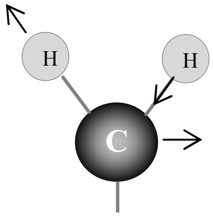
INTRODUCTION

Most of us are quite familiar with infrared radiation, one kind of radiation which can generate heat. The proof is we have seen infrared lamps keep food hot. In fact the heat is associated with the motion and kinetic energy of molecules. The infrared region of the electromagnetic spectrum falls between 14,000 cm^{-1} to 10 cm^{-1}. The region of most interest for chemical analysis is the mid-infrared region (4,000 cm^{-1} to 400 cm^{-1}) Infrared radiation in this range is absorbed and converted by an organic molecule into energy of molecular vibration. By examining how this occurs, we will learn how the study of infrared spectroscopy can provide information about the structure of organic molecules.

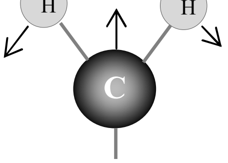
The movement of atoms and bonds is similar to balls connected with springs, as shown in figure below. The major types of molecular vibrations are stretching and bending of bonds. Each organic molecule generates different frequency of vibration.



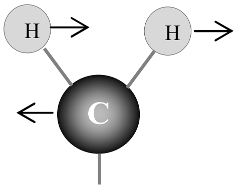
symmetric stretching
(~2850 cm^{-1})



asymmetric stretching
(~2925 cm^{-1})



HCH bend
(~1450 cm^{-1})



rocking motion
(~750 cm^{-1})

The main goal of IR spectroscopic analysis is to determine the chemical functional groups in the sample. Different functional groups absorb characteristic frequencies of IR radiation. In IR spectroscopy, a simple molecule can produce a very complex spectrum. Like fingerprints, IR spectra can be compared with the spectra of known molecules and be used for identification purposes.

MATERIAL AND APPARATUS

acetone, benzene, mixture of acetone:benzene 1:1, NaCl plate, cell holder

PROCEDURE

Handling IR Cells; Liquid Sample Preparation

Remember, the crystals are table salt! DO NOT EXPOSE THEM TO WATER OR BARE FINGERS.

1. Remove the IR cell from the storage desiccator. Carefully place the pieces on a clean cloth.
2. Place 1 or 2 drops of liquid sample on the face of salt plate using dropper. Another salt plate was put on top and squeezed to form a thin, uniform film.
3. Do not use excessive pressure when assembling the IR cell. This will fracture the salt crystals.
4. Place the plate in a holder and ready for analysis.

Measurement

1. Perform a Background Scan.
2. Obtain the IR spectrum of the sample.
3. Remove the holder from the instrument.
4. Separate the crystals cell and clean the faces of cells using a clean cloth. Use acetone on the cloth is necessary. Allow the crystals to air dry.
5. Proceed with the next sample.

RESULTS AND DISCUSSION

On each IR spectrum, **neatly label the major absorption peaks**, indicate the approximate wavenumbers and the functional groups that give rise to the particular IR absorption peaks.

Comparing the IR spectra, mention at least one distinguishing absorption peak for each compound that is absent in the spectrum of the other compound.

In your results and discussion, you should include the below:

1. Why is it essential that you not handle NaCl plates with your bare hands?
2. Which region of the IR spectrum is most useful for identifying specific functional groups in molecules?
3. What is the purpose of background scanning?

CONCLUSION

REFERENCE

APPENDICES A: EXAMPLE OF LAB COVER PAGE

UCSI
UNIVERSITY
The University. The Experience

EP 102 FOUNDATION OF CHEMISTRY I
LABORATORY REPORT

EXPERIMENT 1: WASTEWATER TREATMENT METHOD OF PALM OIL MILL EFFLUENT

TAN JULLY (GROUP NO. 4)

DATE OF EXPERIMENT:
DATE OF SUBMISSION:

SCHOOL OF ENGINEERING
FACULTY OF ENGINEERING, ARCHITECTURE & BUILT ENVIRONMENT

APPENDICES B: REFERENCE

Reference Style

The details of the references cited in the text, published or unpublished should be located in the List of References. The list should be placed at the end of the thesis, a listing of sources actually cited, compiled either alphabetically (Harvard System) or numerically (Number System). The style selected must be used consistently throughout the thesis.

Author and Year System

This system is also known as Harvard System.

Citing in the Text

The references cited in the text should be indicated using the name of the author and the date of publication. Examples are as follow:

(a) If the name of an author is written as part of a sentence, the year published should be written in parentheses.

"Works by Yao (1993) have shown that in order to maintain the behavioural link between the offsprings and their parents, the use of crossover operator should be avoided."

(b) If the name of an author is not written as part of a sentence, both the name and year published should be written in parentheses.

"ANN offers useful properties and capabilities such as non-linearity, input and output mapping, adaptability and fault tolerance among others (Haykin, 1999)."

(c) If there are two authors for a cited reference, both names should be written.

"In designing the model for non-linear system, the parsimonious principle (Soderstrom and Stoica, 1989) is critical because a nonlinear model involves an excessive number of parameters."
"Syu and Chang (1999) successfully used neural networks to adaptively control *Penicillin acylase* fermentation."

(d) If there are more than three authors for a cited reference, use *et al.* after the name of the first author.

"The algorithm can be calculated by applying Gram-Schmidt procedures as described by Korenberg *et al*. (1988)."

(e) If more than one reference materials by the same author in a same year are cited, use small letter alphabets (a, b, c, and so on) to distinguish them.

"Some of the basic principles widely used by many researchers are Lagrange-Euler (LE) equations (Uicker, 1965; Bejczy and Paul, 1981), Newton-Euler (NE) equations (Luh *et al*., 1980a) and d'Alembert (GD) equations (Lee *et al*., 1983)."

"Luh *et al.* (1980b) presented an example of an acceleration control of robot arm/manipulator."

(f) Cross referencing in not allowed in a thesis. Only primary sources should be used.

Writing Style for Authors' Names in the List of References

Generally, authors' names are listed using surname followed by their initials. The followings are examples of writing style according to the name of the author:

(i) Single and multiple authors

Example (single author) :

Veres, S. M. (1990). *Structure Selection of Stochastic Dynamic Systems*. New York:

Gordon and Breach Science Publishers.

Example (two or more authors):

Soderstrom, T., and Stoica, P. (1989). *System Identification*. United Kingdom: Prentice Hall International Ltd.

Luh, J. Y. S., Walker, M. W., and Paul, R. P. (1980b). Resolved-Acceleration Control of Mechanical Manipulators. *IEEE Trans. Automatic Control*. 25(3): 468-474.

(ii) Editor

Example:

Martin, A. M. (Ed.) (1991). *Peat as an Agent in Biological Degradation of Waste*. London: Elsevier.

Lees, R. H. and Thomas T. R. (Eds.) (1974). *Chemical Nomenclature Usage*. Chichester: Ellis Horwood.

(iii) Corporate author/editor

Example:

Engineers Joint Council (1969). *Thesaurus of Engineering and Scientific Terms*. New York: Engineers Joint Council.

Writing Style for Various Types of Publication Materials in the List of References

Frequently, different types of publication materials are cited in a thesis. The style of writing details on cited publication should be as follows:

(i) Book

Author (Year). *Title*. (Edition). Placed published: Publisher.

Example:

Theusen, G. J. and Fabrycky, W. J. (1984). *Engineering Economy*. (6th ed.) Englewood Cliffs, N. J.: Prentice Hall.

(ii) Article in a book

Author of the article (Year). *Title of the article*. In author or editor of the book. *Title of the book*. (page). Place published: Publisher.

Example:

Hussein, S. B., Jamaluddin, H., Mailah, M. and Zalzala, A. M. S. (2000). An Evolutionary Neural Network Controller for Intelligent Active Force Control. In Parmee, I. C. (Ed.) *Evolutionary Design and Manufacturing* (pp. 351 –362). London: Springer-Verlag.

(iii) Journal articles

Printed format

Author (Year). Title of the article. *Title of the Journal*. Volume (Issue no.), page.

Example:

Billings. S. A. (1980). Identification of Nonlinear Systems: A survey. *Proc. IEE*, *Part D.* 127(6), 272-284.

Electronic format

Author (Year). Title of the article. *Title of the Journal*. Volume (Issue no.), page. Publisher.

Example:

Borman, W. C. (1993). Role of Early Supervisory Experience in Supervisor Performance. *Journal of Applied Psychology*, 78, 443 – 449. American Psychology Association.

(iv) Conference articles

Author (Year). Title of the article. *Name of the conference*. Date of the conference. Place, page.

Example:

Sheta, A. F. and De Jong, K. (1996). Parameter Estimation of Nonlinear Systems in Noisy Environments Using Genetic Algorithms. *Proceedings of the 1996 IEEE International Symposium on Intelligent Control*. 15-18 September. Dearborn, Michigan: IEEE, 360 - 365.

(v) Thesis

Author (Year). *Title of the thesis*. Thesis award. Place published.

Example:

Adnan bin Hassan (2002). *On-line Recognition of Developing Control Chart Patterns*. Ph.D. Thesis. Universiti Teknologi Malaysia, Skudai.

(vi) Legislations

Name of the country (year). *Title of the legislation*. Legislation number.

Example:

Malaysia (1983). *Perintah Monumen Lama dan Tapak Tanah Bersejarah*. P.U.(A)41 1983.

(vii) Standards

Name of the institution (Year). *Standard number*. Place published: Publisher.

Example:

British Standards Institution (1990). *B.S. 764.* London: British Standards Institution.

(viii) Patent

Print format

Author (Year). *Patent number*. Place published: Official source.

Example:

Smith, I. M. (1988). *U.S. Patent No. 123, 445.* Washington DC: U.S. Patent and Trademark Office.

Electronic format

Author (Year). *Patent number*. Retrieved on date, year, from URL address of the patent database.

Example:

Ulrich, K. (2001). *European Patent No. EP1162184.* Retrieved on March 7, 2002, from fttp://ep.espacenet.com/

(ix) Brochure

Name of organization (Year). *Title* [Brochure]. Place published: Publisher.

Example:

Research and Training Center (1993). *Guidelines for Reporting and Writing About People with Disabilities.* [Brochure]. Lawrance, KS: Macmillan.

(x) Measured drawings

Author (Year). *Title*. [Measured drawing]. Name of organization: Place published.

Example:

Salim Man (1989). *Pengisi Sekam ke Dalam Kontena Penyimpan: Pandangan Isometrik.* [Lukisan Teknik]. Universiti Teknologi Malaysia: Skudai.

(xi) Unpublished materials

Author (Year). *Title*. Unpublished note, Name of organization.

(xii) Newspaper article

Print format
No author
Title of article. (Year, date). *Name of newspaper*, page.

Example:
Gearing up to meet new challenges. (2000, February 22). *The Star*. p. 2.

With author
Author. (Year, date). Title of article. *Name of newspaper*, page.

Example:
Izatun Shari (2000, April 18). K-economy: draft out in October. *New Straits Times.* p. 2-4.

Electronic format
Author. (Year, date). Title of article. *Name of newspaper*, Retrieved date, year, from URL address of the newspaper.

Example:
Rosmawati Mion (2006, June 17). Sindiket judi haram tumpas. *Utusan Malaysia.* Retrieved June 19, 2006, from http://www.utusan.com.my

(xii) Magazine

Author. (Year). Title of article. *Name of magazine*, Volume/Issue no., page

Example:

Smith, B. L. (1994). Biofeedback. *Science,* 62, 673 – 675.

(xiii) CD-ROM

Author. (Year). Title of article. [CD-ROM]. *Title of Journal,*, Volume, page. Publisher.

Example:

Ivry, R. B. (1995). Perception and production of temporal intervals across a range of durations. [CD-ROM]. *Journal of Experimental Psychology*, 21, 3-18. American Psychological Association.